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CATALYST SYSTEMS CONTAINING CATALYSTS ENCAPSULATED IN WAX  
[KATALYSATORSYSTEME, ENTHALTEND IN WACHS EINGEKAPELTE  
KATALYSATOREN]

MARTIN KREYENSCHMIDT ET AL

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INVENTOR(S)	(72):	MARTIN KREYENSCHMIDT AND EKKEHARD JAHNS
APPLICANT(S)	(71):	BASF AG, LUDWIGSHAFEN, FEDERAL REPUBLIC OF GERMANY
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Description

[0001] The invention concerns catalyst systems having at least one catalyst (c), which catalyzes a reaction of polyisocyanates (a) with compound (b) with at least two hydrogen atoms, which are reactive with respect to isocyanate groups and are encapsulated in wax, as well as a process for their production. The invention also concerns the use of catalyst systems to produce polyisocyanate polyaddition products, in particular elastic, cellular and compact polyurethanes, as well as a process for their production.

[0002] The conversion of polyisocyanates into polyisocyanate polyaddition products, in particular into polyurethanes, is generally carried out in the presence of catalysts. The reaction profile of a polyurethane system is decisively controlled by the type and amount of used catalysts. The type and amount of used catalysts determines, on the one hand, the reaction start after the isocyanate and polyol components are mixed and controls, on the other hand, the demolding time of the molding to be produced. The demolding time describes the time period after which a workpiece can be removed from the mold without being damaged during the process. The demolding

time is decisively determined by the amount of used catalysts and should be as short as possible for economic reasons.

[0003] The amount of catalysts used in a PUR system cannot be increased at will, however, since the catalysis also fundamentally influences the starting times of the systems. The catalyst amount cannot be increased beyond a specific catalyst concentration, which is predetermined by the system and the technical processing mode, since the system would otherwise already react when the components are mixed. A processing is no longer possible in these cases.

[0004] Acid-blocked tertiary amines for the realization of short demolding times are known from the state of the art. An ammonium salt, which is catalytically inactive, is produced through the conversion of a tertiary amine catalyst with acid. The balance between salt and free amine or acid is shifted to the side of the free amines in the reacting PUR system due to the produced reaction heat, and the catalyst is activated. The clear disadvantage of this system is that combinations of amine catalysts are usually present in the PUR systems and different acid-base balances occur between catalysts when only one blocked catalyst is used. The reaction profile of

the entire system is influenced by the change of the balances and it cannot be guaranteed that the processability will remain constant. The dissociation temperatures are frequently so high that the catalyst can be released only to a limited extent. In addition, it is also not possible to block highly effective metalorganic catalysts with this technology. The acid, which is partially released into the system, also leads to corrosion on the processing machine.

[0005] It is now an object of the invention to make available a catalyst system for the production of polyisocyanate polyaddition products, in particular polyurethanes, which influences less the starting times of the system but accelerates the hardening behavior of the materials and thus clearly reduces the demolding times and similar parameters, such as, for example, the flexing times of integral foams.

[0006] It is another object of the invention to make available a catalyst system, which has the aforementioned advantages and comprises metalorganic catalysts.

[0007] The object can be attained by encapsulating in wax a catalyst (c) suitable for the production of polyisocyanate polyaddition products.

[0008] The object of the invention is therefore a catalyst system, which contains at least one catalyst (c), which catalyzes a reaction of polyisocyanates (a) with compounds having at least two hydrogen atoms (b), which are reactive with respect to isocyanate groups, and which is encapsulated in wax.

[0009] Another object of the invention is a process for producing the catalyst system according to the invention.

[0010] It is likewise an object of the invention to use the catalyst system according to the invention to produce polyisocyanate polyaddition products, in particular polyurethane.

[0011] An object of the invention is finally a process for producing isocyanate polyaddition products, which can be obtained by converting

- a) Polyisocyanates with
- b) High molecular compounds with at least two hydrogen atoms, which are reactive with respect to isocyanate groups, in the presence of
- c) Catalysts, and if required
- d) Blowing agents,
- e) Chain extending agents, and
- f) Auxiliary agents or additives.

characterized in that a part or the entire amount of catalyst (c) is encapsulated in wax.

[0012] Under wax are understood within the scope of the invention compounds whose melting point is in general between 20 and 150°C, preferably between 30 and 130°C, and particularly preferably between 40 and 120°C. The waxes of the invention can also be kneaded or can be solid to brittle hard, but not glass-like in the solid state. The waxes of the invention are generally already relatively low viscous and preferably not stringy somewhat above the melting point.

[0013] The term waxes comprises natural waxes, chemically modified waxes, and synthetic waxes. Natural waxes comprise vegetable waxes, such as, for example, montan wax, animal waxes, such as, for example, beeswax, mineral waxes, and petrochemical waxes, such as, for example, petrolatum and microwaxes. Chemically modified waxes comprise, for example, hard waxes, such as

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montanester waxes, synthetic waxes, among others alkane waxes, such as, for example, wax alcohols, in particular high molecular water-insoluble fatty alcohols with preferably more than 12 carbon atoms, such as, for example, lignoceryl alcohol, ceryl alcohol, myricyl alcohol,

melissyl alcohol, and polyalkylene oxides, such as, for example, polyethylene oxide, poly-THE polyvinyl ether waxes, polyolefin waxes, and oxidized polyolefin waxes. The term waxes also comprises fatty acids, preferably with at least 9 carbon atoms, such as, for example, behenic acid, tetracosanoic acid, and cerotic acid, which can be esterified with alcohols if required, and high molecular polyesters with a molecular weight of  $> 1000$  g/mol, preferably 1500 g/mol, which can be obtained by converting dicarboxylic or polycarboxylic acids with 2 to 20 carbon atoms with dialcohols or polyalcohols with 2 to 30 carbon atoms, wherein the corresponding acids or alcohols can contain aliphatic and/or aromatic structural units. Mixtures of the aforementioned waxes can also be used.

[0014] The term polyisocyanate polyaddition products comprises addition products, which contain in general urethane, allophanate, urea, biuret, uretdione, amide, isocyanurate, carbodiimide and/or uretonimine groups. Urethane and urea groups are preferred.

[0015] The polyisocyanate polyaddition products according to the invention can be compact or cellular. In a preferred embodiment, polyisocyanate polyaddition products are soft, hard, semi-rigid, or integral foams



based on polyurethane, or compact, preferably thermoplastic polyurethanes and/or pourable elastomers.

[0016] The catalyst system according to the invention contains a catalyst (c), which catalyzes a reaction of polyisocyanates (a) with compounds having at least two hydrogen atoms (b), which are reactive with respect to isocyanate groups, comprising at least two different catalysts. Catalysts that catalyze a conversion of isocyanate with an OH-functional group into a urethane bond, so-called wetting catalysts, and catalysts that support in particular the conversion of water with isocyanate into carbamic acid, so-called blow catalysts, are preferably used. A mixture of wetting catalysts and blow catalysts is also preferred.

[0017] Organic amines, such as triethylamine, tributylamine, dimethyl benzylamine, N,N,N',N'-tetramethyl ethylene amine, N,N,N',N'-tetramethyl butadiene amine, N,N,N',N'-tetramethyl methyl hexane-1,6-diamine, dimethyl cyclohexylamine, pentamethyl dipropylene triamine, pentamethyl diethylene triamine, 3-methyl-6-dimethyl amino-3-azapentol, dimethyl aminopropyl amine, 1,3-bisdimethyl amino butane, bis-(2-dimethyl aminoethyl)-ether, N-ethyl morpholine, N-methyl morpholine, N-cyclohexyl morpholine, 2-dimethyl aminoethoxy ethanol, dimethyl ethanolanine,

tetramethyl hexamethylene diamine, dimethylamino-N-methyl  
 ethanolamine, N-methyl imidazole, N-(3-aminopropyl)  
 imidazole, N-(3-aminopropyl)-2-methyl imidazole, 1-(2-  
 hydroxyethyl) imidazole, N-formyl-N,N'-dimethyl butylene  
 diamine, N-dimethyl aminoethyl morpholine, 3,3'-bis-  
 dimethyl amino-di-n-propylamine and/or 2,2'-dipiperazine  
 diisopropyl ether, dimethyl piperazine, N,N'-bis-(3-  
 aminoopropyl) ethylene diamine, tris-(N,N-dimethyl  
 aminopropyl)-s-hexahydrotriazine, 1,4-diazabicyclo-[2,2,2]-  
 octane, 4-chloro-2,5-dimethyl-1-(N-methyl aminoacetyl)-  
 imidazole, 2-aminopropyl-4,5-dimethoxy-1-methylimidazole,  
 1-(3-aminopropyl)-2-ethyl-4-methylimidazole, 1-(3-  
 aminopropyl) imidazole and/or 1-(3-amonpropyl)-2-methyl  
 imidazole, tris(dimethyl aminomethyl) phenol, N,N-dimethyl  
 aminoethyl morpholine, N,N,N',N'-tetramethyl-1-butadiamine,  
 1,8-diazabicyclo-5,4,0-undecane, 1,4-ethylene piperidine,  
 N,N-dimorpholino diethyl ether, N,N-dimethyl-N'-2-hydroxy  
 propyl-1,3-propylene diamine, N-butyl morpholine, N,N,N'-  
 trimethyl-N'-hydroxyethyl-bis(aminoethyl) ether, N,N-bis-  
 (3-dimethylamino propyl) amino-2-propanol, and N-methyl-N'-  
 (2-dimethyl amino) ethyl piperazine or mixtures containing  
 at least two of the mentioned amines are taken particularly  
 into consideration, wherein also high molecular tertiary  
 amines, such as, for example, those described in DE-A 28 12

256, are possible. The usual organic metal compounds, preferably organic tin compounds, such as tin(II) salts of organic carboxylic acids, such as, for example, tin(II) acetate, tin(II) octoate, tin(II) ethyl hexoate, and tin(II) laurate, and dialkyl tin(IV) salts of organic carboxylic acids, for example, dibutyl tin diacetate, dibutyl tin dilaurate, dibutyl tin maleate, and dioctyl tin diacetate can be used in addition as catalysts for this purpose. Potassium salts of carboxylic acids, such as, for example, potassium acetate, and metal catalysts, comprising bismuth, iron, or zirconium compounds, such as, for example, iron acetyl acetonate, can also be used.

[0018] Tertiary aliphatic and/or cycloaliphatic amines can preferably be contained in the mixtures, particularly preferably triethylene diamine.

[0019] Mixtures containing catalysts on amine base as well as also catalysts based on organic metal compounds can furthermore be used as catalyst.

[0020] The catalysts (c) are generally dissolved or suspended in heated liquid wax in order to produce the catalyst systems according to invention. A solubilizer, which provides the catalyst with a better dilution in the wax, can be added if required. The mixture of liquid wax and catalyst is now cooled and dispersed preferably in a

polar liquid. A dispersing agent, which leads to an advantageous dispersion, can be added, if required, before this melting step. The dispersion is carried out in such a way in a preferred embodiment that the catalyst system according to the invention is available in the form of particles, including particles with an average particle diameter of 0.5 to 500  $\mu\text{m}$ , preferably from 1 to 100  $\mu\text{m}$ , and more preferably from 2 to 50  $\mu\text{m}$ .

[0021] As polar liquid is suitable in general any liquid, in which the wax is insoluble. Water or (b) a compound with at least two hydrogen atoms, which are reactive with respect to isocyanate groups, is preferably used. As component is preferably used therein a polyol, in particular a polyetherol, particularly preferably the polyol used during the production of polyisocyanate polyaddition products. The utilization of this polyol is advantageous, since no foreign substance can be introduced into the system in this way and a possible destabilization of the foam due to foreign substances can be prevented in cellular products.

[0022] Compounds that can build up, for example, hydrogen bridges into catalysts (c) and are able to improve the solubility in this way can be used in general as solubilizers. Polar polymers, such as, for example,

polyethylene oxide or polypropylene oxide or mixed polyethers of ethylene oxide and propylene oxide, are taken into consideration, for example. The solubilizer is usually used in an amount of 0.1 to 10% by weight, preferably 0.5 to 5% by weight, with reference to the weight of wax and catalyst (c).

[0023] Amphiphilic molecules, which have a hydrophobic part that is compatible with the wax, and a hydrophilic part that is compatible with the polar liquid, are suitable in general as dispersing agents. Stearic acid, for example, can be used for this purpose. The dispersing agent is usually used in an amount of 0.1 to 20% by weight, preferably 1 to 10% by weight, with reference to the weight of wax and catalyst (c).

[0024] In another embodiment for the production of catalyst systems according to the invention, the liquid mixture of catalyst (c) and heated wax can be brought directly into a suitable particulate form by means of spraying within an air current, preferably a cold air current, so that a preferably granular powder is obtained. From the point of view of the technology is suitable an apparatus similar to a spray dryer, which is however operated with a cold air current. The catalyst system,

which is dispersed within the polar liquid, can also be freeze dried. The catalyst dispersion is first frozen herein and the dispersing agent is removed in a vacuum. A dry powder is obtained. Both processes lead preferably to powders containing particles with an average particle diameter of 0.5 to 500  $\mu\text{m}$ , preferably 1 to 100  $\mu\text{m}$ , and more preferably 2 to 50  $\mu\text{m}$ .

[0025] The catalyst systems according to the invention can be advantageously utilized for the production of polyisocyanate polyaddition products, in particular polyurethanes. The catalyst systems according to the invention make possible to adjust the reaction profile, for example, of a PUR system according to need. The catalysts (c) encapsulated in the wax hardly influence the start times of the system. Heat is released, however, during the course of the reaction, the wax melts starting at a specific temperature, and releases the catalyst. Depending on the melting point of the used wax, it is possible to achieve the release of the catalyst at a different time point of the reaction coordinates. The increased catalyst concentration leads in general to clearly shorter demolding times.

[0026] 100% of the catalyst system according to the invention can consist of catalysts encapsulated in wax.

However, smaller amounts can already show the aforementioned effects, that is, the catalyst system can also contain other catalysts, which are not encapsulated in wax, in addition to the catalyst (c) encapsulated in wax. The weight proportion of catalyst encapsulated in wax with reference to the overall weight of the used catalyst (c) amounts in general to 5 to 100%, preferably 10 to 100%, even more preferably 20 to 100%, and especially 50 to 100%.

[0027] In the process according to the invention for the production of polyisocyanate polyaddition products is used a catalyst (c), wherein a part or the total amount of catalyst (c) is encapsulated in wax, that is, a conventional catalyst, which is not encapsulated in wax, can also be used in addition to the catalyst system according to the invention. The weight proportion of the catalyst encapsulated in wax in the process of the invention amounts to 5 to 100%, preferably 10 to 100%, even more preferably 20 to 100%, and especially 50 to 100%, with reference to the total weight of the used component (c).

[0028] The catalyst system according to the invention comprises in general 5 to 95% by weight, preferably 50 to 90% by weight of catalyst, and 5 to 95% by weight, preferably 10 to 50% by weight of wax, with reference to the overall weight of the catalyst system.

[0029] The used polyisocyanates include the aliphatic, cycloaliphatic, and aromatic isocyanates known from the state of the art. Examples of these are 4,4'-diphenyl methane diisocyanate, mixtures of monomeric diphenyl methane diisocyanates, and highly granular homologues of diphenyl methane diisocyanate (polymer MDI), tetramethylene diisocyanate, tetramethylene diisocyanate trimers, hexamethylene diisocyanate, hexamethylene diisocyanate trimers, isophorone diisocyanate trimer, 4,4'-methylene bis-(cyclohexyl) diisocyanate, xylylene diisocyanate, tetramethyl xylylene diisocyanate, dodecyl diisocyanate, lysine alkyl ester diisocyanate, wherein alkyl represents C<sub>1</sub> to C<sub>10</sub>, 2,2,4- or 2,4,4-trimethyl-1,6-hexamethylene diisocyanate 1,4-diisocyanatocyclohexane, 1,3- or 1,4-bis(isocyanatomethyl) cyclohexane or 4-isocyanatomethyl-1,8-octamethylene diisocyanate.

[0030] Polyisocyanates with NCO groups of different reactivity, such as 2,4-toluylene diisocyanate (2,4-TDD), 2,4'-diphenyl methane diisocyanate (2,4'-MDI), triisocyanatotoluol, isophorone diisocyanate (IPDI), 2-butyl-2-ethyl pentamethylene diisocyanate, 2-isocyanatopropyl cyclohexyl isocyanate, 3(4)-isocyanatomethyl-1-methyl cyclohexyl isocyanate (H-TDI) are likewise preferably used.



[0031] 4,4'-diphenyl methane diisocyanate, 1,3- and 1,4-phenylene diisocyanate, toluylene diisocyanate, isophorone diisocyanate (IPDI), and naphthylene diisocyanate (NDI) are preferably used as polyisocyanates. 4,4'-MDI is especially preferably used.

[0032] Mixtures of the aforementioned isocyanates are preferably used as polyisocyanates.

[0033] Compounds carrying two or more reactive groups, selected from among OH groups, SH groups, NH groups,  $\text{NH}_2$  groups, and CH acid groups, such as, for example,  $\beta$ -diketo groups, in the molecule are taken especially into consideration as compounds having at least two hydrogen atoms (b), which are reactive to isocyanate groups.

[0034] Those with a functionality of 2 to 8, preferably 2 to 6, and a molecular weight of 300 to 8000, preferably 400 to 4000, are suitably used. Polyether polyamines and/or preferably polyols selected from among the group of polyether polyols, polyester polyols, polythioether polyols, polyester amides, polyacetals containing hydroxyl groups, and aliphatic polycarbonates or mixtures of at least two of the mentioned polyols have been proven and tested. Polyester polyols and/or polyether polyols are preferably utilized. The hydroxyl number of

polyhydroxyl compounds amounts therein as a rule to 10 to 1000 and preferably 20 to 300.

[0035] In a preferred embodiment, the compounds (b) having at least two H-atoms that are reactive with respect to isocyanate groups are polyether polyols. They are produced according to known processes, for example, by means of anionic polymerization with alkali hydroxides or alkali alcoholates as catalysts and adding at least one starter molecule, which contains 2 to 3 bonded hydrogen atoms, from one or several alkylene oxides with 2 to 4

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carbon atoms in the alkylene radical. Suitable alkylene oxides are, for example, tetrahydrofuran, 1,3-propylene oxide, 1,2- or 2,3-butylene oxide, and preferably ethylene oxide and 1,2-propylene oxide. The alkylene oxides can be utilized separately, alternatively one after the other, or as mixtures. Mixtures of 1,2-propylene oxide and ethylene oxide are preferred, wherein the ethylene oxide is used in amounts of 10 to 50% as ethylene oxide end cap ("EO cap"), so that the produced polyols have up to more than 70% primary OH end groups.

[0036] As starter molecule are taken into consideration water or bivalent or trivalent alcohols, such as ethylene glycol, propandiol-1,2 and -1,3, diethylene glycol,

dipropylene glycol, butandiol-1,4, glycerin, trimethylol propane, et cetera. Polyether polyols, preferably polyoxypropylene polyoxyethylene polyols, have a functionality of 2 to 3 and molecular weight of 1000 to 8000, preferably 2000 to 7000.

[0037] As polyetherols are also suitable polymer modified polyether polyols, preferably graft polyether polyols, in particular those based on styrene and/or acryl nitrile, which are produced by means of in situ polymerization of acryl nitrile, styrene or preferably mixtures of styrene and acryl nitrile.

[0038] Polyester polyols are also suitable. These can be produced, for example, from organic dicarboxylic acids with 2 to 12 carbon atoms, preferably aliphatic dicarboxylic acids with 4 to 6 carbon atoms, polyvalent alcohols, preferably diols, with 2 to 12 carbon atoms, preferably 2 to 6 carbon atoms. As dicarbonic acids are taken especially into consideration: succinic acids, glutaric acid, adipic acid, suberic acid, azelaic acid, sebacic acid, decane dicarboxylic acid, maleic acid, fumaric acid, phthalic acid, isophthalic acid, and terephthalic acid. Dicarbonic acids can be utilized therein separately or in mixtures among each other. Instead of the free dicarboxylic acids can also be used the

corresponding dicarboxylic acid derivatives, such as, for example, dicarboxylic acid esters of alcohols with 1 to 4 carbon atoms or dicarboxylic acid anhydrides. Dicarboxylic acid mixtures of succinic, glutaric, and adipic acid and aromatic diacids are preferably used. Examples of bivalent and polyvalent alcohols, in particular diols, are: ethandiol, diethylene glycol, 1,2- or 1,3-propandiol, dipropylene glycol, 1,4-butandiol, 1,5-pentandiol, 1,6-hexandiol, 1,10-decandiol, glycerin, and trimethylol propane, and also dialcohols containing aromatic or aliphatic ring systems, such as, for example, 1,4-bis(dihydroxy methyl benzol, or 1,4-bis(dihydroethyl benzol. Ethandiol, diethylene glycol, 1,4-butandiol, 1,5-pentandiol and 1,6-hexandiol are preferably used. Polyester polyols from lactones, for example,  $\epsilon$ -caprolactone or hydroxy carboxylic acids, for example, hydroxy caproic acid, can also be used. Mixed systems containing polyesterols as well as also polyetherols can likewise be utilized.

[0039] The use of blowing agents is also necessary for the production of cellular polyisocyanate polyaddition products. Water is preferably used as blowing agent (d). Since the components (b) and if required (e) can have water as a result of the production and/or chemical composition, a separate addition of water to the components (b) and (e)

or the reaction mixtures is necessary in many cases. Insofar as water must be added to the polyurethane formulation in order to obtain the desired spatial weight, this water is usually added in amounts of 0.05 to 4.0% by weight, preferably 0.1 to 3.0% by weight, and in particular 0.3 to 2.5% by weight, with reference to the weight of the components (a) and (b) and if required (c).

[0040] Low boiling liquids, which evaporate under the influence of the exothermal polyaddition reaction and have a boiling point under normal pressure within the range of -40 to 120°C, preferably 10 to 90°C, or gases can also be used as blowing agents (d) instead of water or preferably in combination with water. Of the liquids suitable as blowing agents (d) that are inert with reference to NCO groups are preferably utilized alkanes, cycloalkanes, or mixtures with a boiling point of -40 to 50°C under atmospheric pressure from alkanes and cycloalkanes.

[0041] The liquids of the kind mentioned above and gases that are suitable as blowing agents can be selected, for example, from the group of alkanes, such as, for example, propane, n-butane or isobutane, n-pentane or isopentane, and preferably technical pentane mixtures, cycloalkanes, and cycloalkenes, such as, for example, cyclobutane, cyclopentene, cyclohexane, and preferably

cyclopentane and/or cyclohexane and gases, such as, for example, nitrogen, carbon monoxide, and noble gases, such as, for example, helium, neon and krypton.

[0042] Salts, which are thermally degradable, such as, for example, ammonium bicarbonate and/or ammonium carbonate or compounds that form such salts in situ, such as, for example, aqueous ammonia and/or amines and carbon dioxide, and ammonium salts of organic carboxylic acids, such as, for example, monoammonium salts of malic acid, boric acid, formic acid, or acetic acid, are also suitable as blowing agents. HFC-245fa® (Honeywell Company) and HFC365mfc® (Solvay Company) can likewise be used.

[0043] The chain extending agents (e), which may be utilized in the invention, if required, can function as chain extending agents, but also as wetting agents, comprise low molecular, polyvalent alcohols, preferably diols and/or thiols, with molecular weights of less than 400 Da, preferably 60 to 300 Da, particularly preferably 60 to 200 Da. Aliphatic, cycloaliphatic and/or araliphatic diols, such as, for example, alkandiols with 2 to 14, preferably 2 to 6 carbon atoms and/or dialkylene glycols with 4 to 8, preferably 4 to 6 carbon atoms, such as, for example, ethylene glycol, propandiol-1,3, decandiol-1,10, o-, m-, p-dihydroxy cyclohexane, diethylene glycol,

triethylene glycol, dipropylene glycol, tripropylene glycol, and preferably butandiol-1,3, butandiol-1,4, hexandiol-1,6 and bis-(2-hydroxyethyl) hydrochinone, pentanediol-1,5 and pentanediol-1,3 or mixtures of different diols and as polyvalent alcohols, for example, triols, such as, for example, 1,2,4-, 1,3,5-trihydroxy cyclohexane, trimethylol ethane, glycerin, and trimethylol propane, are taken into consideration, for example. Diols and their mixtures are preferably utilized.

[0044] Auxiliary agents and additives can be added, if required, in the process according to the invention in order to produce polyisocyanate polyaddition products (f).

[0045] As auxiliary agents and additives should be mentioned, for example, surface active substances, foam stabilizers, cell regulators, outer and inner separating agents, fillers, and reinforcing agents, such as, for example, glass fibers, dyes, pigments, flame retardants, hydrolysis stabilizers, oxidation protective agents, abrasion resistance conditioners, fungistatic and bacteriostatic acting substances.

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[0046] Other data about the utilized starting substances can be found, for example, in the Kunstoffhandbuch (Plastics Reference Book), Volume 7,

"Polyurethanes," published by Guenter Oertel, Carl-Hauser-Verlag, Munich, 3<sup>rd</sup> edition, 1993.

[0047] The polyisocyanates (a) with (b) compounds having at least two hydrogen atoms, which are reactive with respect to isocyanate and, if required, (e) chain extending agents are converted in such amounts in order to produce the polyisocyanate polyaddition products that the equivalence ratio of NCO groups of (a) polyisocyanates to the sum of the reactive hydrogen atoms of the components (b) and if required (e) amounts to 1:0.5 to 1:3.50 (corresponding to an isocyanate index of 50 to 350), preferably 1:0.65 to 1:1.30, and particularly preferably of 1:0.9 to 1:1.15.

[0048] 0.01 to 15% by weight, preferably 0.1 to 8% by weight, particularly preferably 0.5 to 4% by weight of catalyst (c) are generally utilized with reference to the weight of the components (a) and (b) and if required (c).

[0049] The polyisocyanate polyaddition products are usually produced according to the known one-shot or likewise known prepolymer processes. A prepolymer is usually produced in a first step from (a) and (b) in the known prepolymer process, preferably a prepolymer containing isocyanate groups, which is then converted with more (b) into the desired products.



[0050] The initial components are usually mixed at a temperature of 0 to 100°C, preferably 15 to 70°C, and brought to react, for example, in a molding tool. The mixing can be carried out mechanically by means of low pressure technology or high pressure technology, or by means of other mixing processes, which are used in customary PUR processing machines.

[0051] The polyisocyanate polyaddition products produced according to the process according to the invention can be used as moldings.

[0052] The catalyst systems according to the invention are preferably used with PU systems, where demolding and flexing times represent a critical parameter or, where long flow paths are required, so that catalysts that become active only at the end of the reaction offer great advantages, since a crosslinking, which can limit the flowability, occurs only as late as possible. Suitable PU systems are, for example, soft, soft integral, semi-rigid, and hard foam.

[0053] The invention will be described with reference to the following examples.

#### Examples

##### Example 1

##### Production of Catalyst Systems

[0054] 150 g of alkane wax with melting point of 100°C, 11 g of stearic acid, and 100 g of DABCO were provided. The preparation was molten while stirring at 120°C. 609 g of polyetherol Lupranol® 2043 were added to the melt and these were dispersed at 90°C with a dissolver (5 cm diameter) at 6000 rpm. The mixture was rapidly cooled to room temperature after 2 minutes by means of further dispersion and a dispersion of crystalline soft parts (melting point approx. 80°C) with an average particles size of about 20 µm in polyetherol was obtained.

#### Examples 2 to 4 and Comparative Examples 5 and 6

##### Production of PU Foams

[0055] The polyol components listed in Table 1 (all data are in weight parts) were converted into foams with an isocyanate component having the following composition, and in doing so were obtained the reaction data shown in Table 2:

Prepolymer based on

54 parts by weight of 4,4'-MDI

44 parts by weight of a polyol with OH number of 28 and average functionality of two, made up of approx. 80%

propylene oxide having a 20% ethylene oxide end cap

2 parts by weight of dipropylene glycol

NCO content: 14%

TABLE 1

Components Polyol Components	Ex. 2	Ex. 3	Ex. 4	Compar- ative Example	Compar- ative Example 6
Polyether with OHZ 28 and average functionality 2, 80% PO and 20% EO end cap	45	45	45	45	45
Polyether with OHZ 26 and average functionality 3.78% PO and 22% EO end cap	36	35.60	35.30	37	37
Butandiol-1,4	14.15	14.15	14.15	14.15	14.15
Water	0.80	0.80	0.80	0.8	0.80
Stabilizer DC 193® (Air-Products)	0.50	0.50	0.50	0.5	0.50
Catalyst system of example 1	1.30	2.00	4.00	0	0
Catalyst Lupragen®	2.50	2.50	2.50	2.5	4.90

N202					
Catalyst Lupragen® N206	0.40	0.40	0.40	0.4	0.40
Catalyst OTS 17D® (AKCROS)	0.05	0.05	0.05	0.05	0.05
Mixing ratio A:B  (with reference to weight parts)	1:1.40	1:1.41	1:1.41	1:1.39	1:1.41

[0056] The following values were obtained during testing:

TABLE 2

Procedure	Ex. 2	Ex. 3	Ex. 4	Compar- ative Example	Compar- ative Example 6
Mixing time [sec]	9	9	9	9	5
Start time [sec]	14	14	14	14	8
Rising time [sec]	53	48	42	71	40
Gelling time [sec]	36	34	30	39	27
Demolding time [sec]	120	120	120	120	120
Flexing time [sec]	210	185	170	240	165
Water proportion	0.1855	0.1855	0.1856	0.1856	0.1854

Plate thickness [g/dl]	565	552	560	543	549
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[0057] The components were mixed with an agitator in the examples 2 to 4 and the comparative examples 5 and 6. The starting, rising, and gelling times were determined with a format rising height measuring device or ultrasound rising height measuring device. The demolding time indicates the time after which a foamed plate is removed from the mold. The flexing time indicates the time after which the demolded foamed plate can be buckled by 180°C, without producing cracks.

[0058] None of the catalysts systems according to the invention was utilized in comparative example 5, and likewise in comparative example 6, where however double the amount of conventional catalyst Lupragen® N202 was used.

[0059] The examples show clearly that short flexing times as well as also high starting times can be achieved only by using the catalyst system according to the invention (examples 2 to 4). The comparative examples 5 and 6 have a high flexing time or a short starting time.

#### Patent Claims

1. A catalyst system, containing at least one catalyst (c), which catalyzes a reaction of polyisocyanates (a) with

compounds (b) having at least two hydrogen atoms, which are reactive with respect to isocyanate groups, and is encapsulated in wax.

2. The catalyst system of claim 1, containing wetting catalysts and/or blow catalysts as catalysts.

3. The catalyst system of claim 1 or 2, which contains a wax having a melting point of 20 to 150°C.

4. The catalyst system of one of the claims 1 to 3, which is available in form of particles with an average particle diameter of 0.5 to 500 µm.

5. A process for producing the catalyst systems of one of the claims 1 to 4, characterized in that the catalysts (c) are accommodated in molten wax and are then dispersed in a polar liquid.

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6. The process of claim 5, characterized in that the polar solution contains water and/or compounds (b) with at least two hydrogen atoms, which are reactive with respect to isocyanate groups.

7. The process for producing the catalysts systems of one of the claims 1 to 4, characterized in that the catalysts (c) are accommodated in molten wax and are then brought into particulate form by means of spraying in an air current or by means of freeze drying.

8. A use of the catalyst systems of one of the claims 1 to 4 for the production of polyisocyanate polyaddition products.

9. The process for the production of polyisocyanate polyaddition products by converting

- a) Polyisocyanates with
- b) High molecular compounds with at least two hydrogen atoms, which are reactive with respect to isocyanate groups, in the presence of
- c) Catalysts, and if required
- d) Blowing agents,
- e) Chain extending agents, and
- f) Auxiliary agents or additives.

characterized in that a part or the entire amount of catalyst (c) is encapsulated in wax.

10. A use of polyisocyanate polyaddition products produced according to claim 9 for the production of moldings.